



Packaging and Reliability of Electronic Nose for Space Applications

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JPL

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JPL Definition

- ◆ Electronic noses are systems for the automated detection and classification of odors of simple and complex and it is generally composed of an array of chemical sensing systems and a pattern recognition system. Electronic noses are based on human olfactory system. (Gardner, 1988)
- ◆ Arrays of sensors, which respond to a wide range of compounds. (Chemical sensors, spectrometers)
- ◆ Advanced pattern recognition and artificial intelligence techniques, which enables to extract relevant and reliable information.(statistical methods such as principal component analysis, discriminant factorial analysis, cluster analysis or artificial neural networks)





Application of an E-Nose

• E-Nose instrument is one which involves the initial inspection of a known set of m different odours and then attempts to identify an unknown odour from a database.



SENSES

- ◆Smell (Nose, Odor, good, bad, etc)
- ◆Taste (Tongue, sweet, spice, sour, etc.)
- ◆Feel (Touch, hard, soft, warm, cold)
- ◆Listen (Ears, sound)
- ◆See (Vision, Eyes, colors)







Objectives

- ◆ Develop, build, and demonstrate a miniature gas sensor, an E-Nose, for experiment on a shuttle flight (Accomplished by JPL)
- ◆ Assess the air quality monitor in crew habitat on a spacecraft
- ◆ Assess and validate various e-noses
- ◆ Establish damage process and failure of e-nose
- ◆ Early failure mechanism by environmental storage and thermal cycling tests
- ◆ Packaging Reliability and E-Nose reliability



Introduction



- ◆ Electronic Nose, New Technology Infusion on Space Shuttles, International Space Station (ISS)
- ◆ Demonstrated the operation of polymer based sensors based on Dr. Nathan Lewis of Caltech for use in sensing arrays
- ◆ E-Nose was flown on STS-95 to determine its utility as an air quality/incident monitor in crew habitat on a spacecraft
- ◆ JPL has developed, built and demonstrated a low power, miniature gas sensor which has capability to identify various gas species present in the recirculated breathing air of the space shuttle.



Introduction



- ◆ Constituents of breathing air in a closed chamber in which air is recycled (Space station, space shuttle, etc.)
- ◆ Air-quality is determined by collecting the samples and analyzing them on the ground using GC-MS
- ◆ Miniaturized e-nose capable of identifying contaminants in the breathing environment at ppm or ppb levels would greatly enhance the capability for monitoring the quality of recycled air as well as providing notification of presence of dangerous gases.
- ◆ An e-nose is an array of non-specific chemical sensors, controlled and analyzed electronically, which mimics the action of nose by recognizing patterns of response to vapors
- ◆ JPL/NASA needs such e-noses



Rationale to NASA

- ◆ No miniature gas sensors capable of detecting and identifying a broad suite of compounds
- ◆ GC-MS are capable of highly accurate analysis at very low levels require significant crew time to operate and maintain
- ◆ Lack of a monitor in crew quarters of space shuttle or space station
- ◆ Need of such capability is foreseen
- ◆ Air in the space station cannot be easily replaced
- ◆ Contaminants will build up over time
- ◆ Air quality should be monitored since crew health is important
- New technology infusion to create an integrated environmental monitoring and control system for the ISS

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- ◆Conductivity sensors (metal oxide and polymer)
- ◆Piezoelectric sensors
- **◆**MOSFET
- **♦**Optical
- **♦** Spectrometry
- **◆**Electrochemical
- **♦**Chemical



Metal-Oxide Sensors

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- **♦** Tin
- ◆ Zinc
- ◆ Tungsten
- **♦** Titanium
- ◆ Iridium

Dopants of Platinum or Palladium

Type of Odor such as CO, CO₂, NH₃

Selectivity as a function of temperature

Water vapor

Humidity





- * Sensors saturate at some value
- Detection limit is determined by the baseline noise the detection limit is often defined as the concentration which yields a response which is two standard deviations above baseline noise
- * Individual sensors are reproducible in their response to a given odor or chemical.
- * The reproducibility of a given sensor is important during the life of the sensor. This reliability/reproducibility is particularly acute for electronic nose.





Metal-oxide gas sensors

The most widely used material is tin-oxide doped with small amounts of Pd or Pt and other oxide materials such as ZnO, TiO₂, WO₃ and perovskites.

Tin oxide resistive sensors have been developed for a range of applications by changing the choice of catalyst and operating conditions

The significant advantage of metal-oxide sensors for many applications is their high sensitivity sub ppm levels for some gases.







Metal Oxide Sensors

Disadvantage: Drift over periods of hours to days, mitigate this using signal-processing algorithms

Poisoning: Poisoning of these sensors by sulfur compounds present in the odor mixture

JPL Chemical Sensor Materials

- NEPP
- Inorganic crystalline or polycrystalline Semiconductors (MOSFET, metal oxides, zeolite absorbent materials, and metallic catalysts)
- Organic materials or polymers advantage of being more flexible in design and more readily modified chemically to develop arrays of materials with different properties (this has lead to electronic nose development)
- Biologically derived materials

 Proteins, enzymes, and antibodies, which is an important component of biosensors. These difficult to stabilize and, to date, have not been widely used as gas sensors or in electronic noses.





Conducting polymer gas sensors

Conducting polymers have attracted much interest as sensor materials for use in electronic noses for several reasons

- ♦ A wide range of materials can be synthesized
- ◆ They respond to a broad range of organic vapors
- ◆ They operate at room temperatures





Conducting polymer gas sensors

Conducting polymer systems have advantages over metal-oxide or other devices

The outstanding problems which need to be addressed are the sensitivity to humidity and the long-term drift.

Ref.: Gardner and Bartlett

Conducting Polymers

- **◆** Polypyrroles
- **♦** Thiophenes
- **♦** Indoles
- **♦** Furans
- Conducting polymers operate at ambient temperature and do not need heaters.
- Conductivity of these materials change when they are exposed to chemicals, which bond with polymers via ionic or covalent

They are suitable for portable instruments







Acoustic wave gas sensors

- Bulk acoustic wave (BAW) devices
- Surface acoustic wave (SAW) devices

Substrates are piezoelectric

Quartz, lithium niobate or zinc oxide

Use soft and rubbery polymers for fast response

Low glass-transition temperature polymers are suitable

Poly(siloxanes) have very low glass-transition temperature (<10°C)

Ref.: Gardner and Bartlett





Surface Acoustic Wave Devices

◆ The SAW device comprises a relatively thick plate of piezoelectric material with interdigitized electrodes, usually of gold, to excite the oscillation of the surface. It relies on the transmission of a surface wave, corresponding to a deformation of the crystal normal to the crystal surface, across the surface of the device. The energy of this device is confined largely to a layer about one acoustic wavelength thick (~100 μm) at the crystal surface.

Piezoelectric Sensors

- ◆ Quartz crystal microbalance (QCM)
- ◆ Surface acoustic wave (SAW)
- These devices can measure

Temperature

Mass changes

Pressure

Force

Acceleration

In electronic nose

they are used as mass-change sensing devices

- ◆ SAW is less sensitive than QCM
- ◆ SAW operates in 100s of MHz and QCM operates at 10s of MHz
- ◆ Cost of SAW is cheaper than QCM
- ◆ Complex electronic circuitry
- ◆ Resonant frequency drifts as the active membrane ages

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MOSFETs

◆ Metal-Oxide-Silicon Field-Effect-Transistor (MOSFET) odor sensing devices are based on the principle that VOCs in contact with a catalytic metal can produce a reaction in the metal. The reaction's products can diffuse through the gate of a MOSFET to change the electrical properties of the device







Field Effect Gas Sensors

Field-effect gas sensors are based on metal-insulator -semiconductor structures in which the metal gate is a catalyst for gas sensing. Typical catalytic metals used in these applications are Pt, Pd, and Ir. There are two basic configurations; the-metal insulator-semiconductor -field -effect-transistor (MISFET) and the metal-insulator-semiconductor capacitor (MISCAP).

Effect of temperature
Effect of catalyst
Effect of continuous and discontinuous gate
Detection limit
Response time





Optical Sensors

◆ Optical-fiber sensors utilize glass fibers with a thin chemically active material coating on their slides or ends. A light source at a single frequency is used to interrogate the active material, which in turn responds with a change in color to the presence of the VOCs to be detected and measured. Arrays of these devices with different dye mixtures can be used as sensors for an electronic nose.





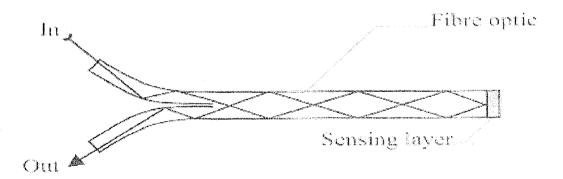


Fiber-optic gas sensors

Fiber-optic gas sensors rely on the light guiding properties of the optical fiber to carry the light from the light source to the chemically sensitive layer and then to return the light to the sensor

Attractive approach to the fabrication of miniature sensor arrays Response can be measured using video technology Remote measurements can be made

No electrical interference



Fiber-optic gas sensor

Electrochemical Gas Sensors





Electrochemical gas sensors are based on the electrochemical Oxidation or reduction of the analyte gas at a catalytic electrode surface. CO, H₂S, NO_x, Cl₂, SO₂, HCN, O₂ The concentration of gas is determined by measuring the current flowing in the sensors

Advantages

Operate at room temperature

Have low power consumption

Robust, reasonably selective

Working life of ~2 years

Detection limit: 0.1 ppm

Response time: 10 s, Reliability, Ruggedness

Uses in personnel protection monitoring in mining, tunneling,

and other industrial applications

Disadvantages

1 - 3 cm in diameter, size

Array will be too bulky

High selectivity for a limited number of simple gases



Pellistors





- Pellistors are detectors for combustible (flammable) gases by detecting the heat liberated when the combustible gas reacts with atmospheric oxygen on the surface of a small catalytic bead.
- Non-specific in their response to combustible gases and respond reasonably rapidly ~20s (500 ppm), operating life time: Several years
- Si, P, and Pb are poisons, high power consumption (350 mW), operate at elevated temperatures, not ideal for use in arrays since limited number of different catalyst types
- Microfabrication techniques to reduce power and size better suited for e-nose

Catalyst bead Platinum wire	
-(00000)-	
2 min	≥ 6 11

Reference.





Physical sensors

- Chemical sensors are influenced by temperature and humidity.
- The effect of temperature and humidity on different types of odour sensors is a significant problem.
- It is highly desirable to control both the temperature and the humidity of the analyte and the operating temperature of the odour sensor.
- Some cases it may not be possible to control these parameters.
- Sensor signals should be compensated for by neural networks
- Measure temperature of the odour





Sensor Arrays

- There is no ideal gas sensor type for use in an electronic nose.
- Sensors specifically for use in arrays
- Single sensor arrays, simplicity of instrumentation, sample preparation.
- Too restricting: single sensor type, single sensing principle, respond to limited range of molecular parameters.
- Consider mixed sensor arrays comprising different types of sensors such as metal oxides, conducting polymers, SAW devices, MOSFETs for future prospects of electronics noses





Mixed Sensor Arrays & Complexity

- Different types of measurement circuits
- Differences in operating temperatures
- Ideal flow conditions
- Sensitivities
- Response times for different sensor types
- Miniaturizing the sensor arrays
- Tin oxide sensor arrays occupy a significant volume because of the size of individual sensor elements. This is a problem when the sample volume is limited
- Some sensor types are more readily miniaturized than others.





Other types of odour sensors

- Chemoresistor with phthalocyanin-based gas sensors
- They have high sensitivity but they only respond to a limited set of reactive gases
- Chemoresistor sensor is based on the effect of solvent swelling on the resistance of carbon-black loaded polymer films.





Long-term stability

- Successful operation of an electronic nose is its long-term stability
- How its baseline and steady-state response vary with time?
- Some sensors require a stabilization period after they have been manufactured or after they have been left unused
- Seasonal and environmental drift in the behavior of e-nose
- Contamination and poisoning of the sensor material
- Silicones and sulfur compounds for semiconducting oxides and strong acids for conducting polymers







Reliability Issues

- It is not worth spending several weeks training the system to recognize complex mixtures for a particular application
- Common problem encountered with reproducibility of response over time arise from drift in the sensitivity of the sensors and effect of poisoning. Drift may be due to the aging effects.
- · Poisoning of sensing material leading to a reduction or even total loss of sensitivity.
- Sensing materials are sufficiently characterized and types of likely poisons identified.
- Poisoning is a far greater problem in applications where a wide variety of different sample types and interfering compounds exist.

JPL Reliability Issues



- Response reproducibility between sensors of nominally the same type. This is important for two reasons
- If a sensor is poisoned then it can be replaced without the need to fully recalibrate and retain the system.
- If the sensors are sufficiently reproducible in their response it becomes possible to train one sensor array and then use this same training set for any number of nominally identical arrays in other instruments at different locations.
- Sensitivity to changes in temperature, humidity, and flow rate are also important characteristics. Odour sensors should be insensitive to changes in temperature, humidity, and flow rate. Most sensors show some sensitivity problem.

JPL Reliability Issues



- These effects can be minimized by careful system design and sample handling which makes the instrument more complex, expensive, and limit sample throughput.
- In portable electronic noses it will be essential to overcome, or at least adequately compensate for, the effects of changes in temperature and humidity on the sensors baseline and on the magnitude of their responses.
- Sensor physical size need to be kept low. If the sensor is large then sample volume is large, which can lead to slow response time and poor sensitivity because of dilution.
- Microfabricated sensor array can meet these requirements.





Reliability

- Avoid polymeric tubing
- If possible SS tubing
- Tests with Teflon?





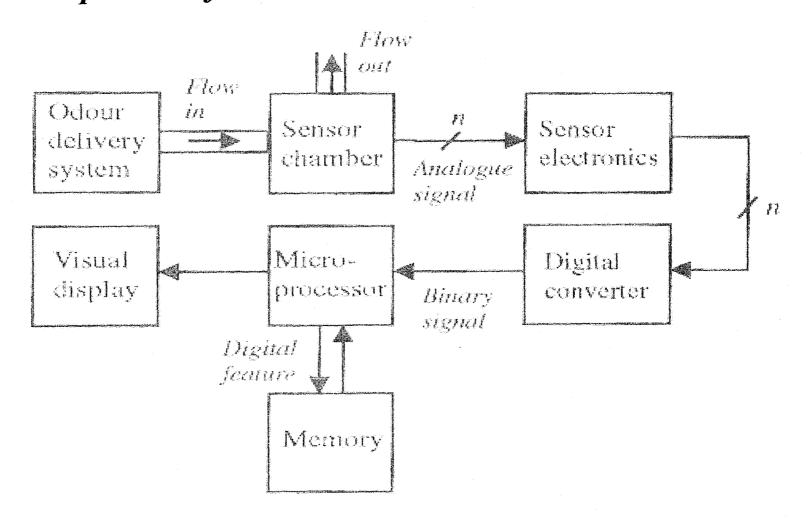
Response Time

- ◆ The response time of a sensor is defined as the time taken for a device to reach some predetermined fraction of its final output in response to a step change in concentration
- ◆ It is necessary that the sensors have an adequate dynamic range and do not saturate in their response in the normally encountered concentration range.
- ◆ The sensors should have a linear response to concentration in order to allow the application of linear data processing techniques.





Basic components of an electronic nose instrument system

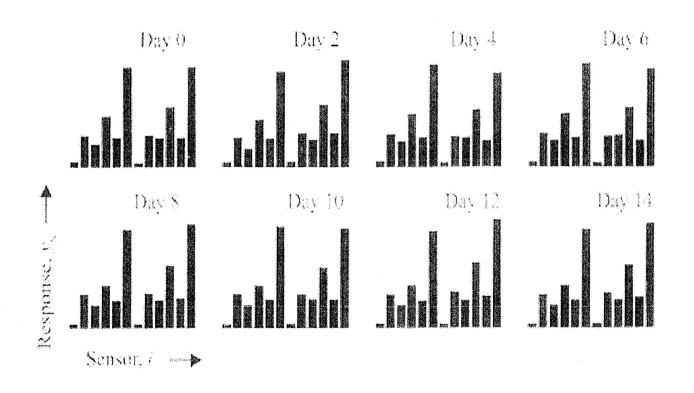


Ref.: Gardner and Bartlett





Repeatability of the response of a Fox 2000 12 MOS Sensors, Heptanol, 2 weeks duration

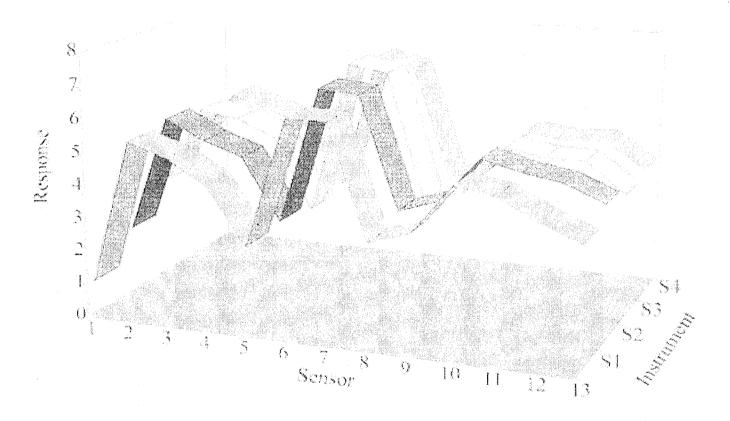


Ref.: Tan et al., 1995





Reproducibility of a set of e-nose 4000 employing conducting polymer chemoresistors



Ref.: Warburton and Hathcock, 1996





Hybrid electronic noses

- Modules of different sensor technologies such as conductometric, BAW, SAW
- Olfactory binding proteins
- Interfering gases
- Poisoning of sensors
- Temperature interference
- Humidity interference
- Battery powered miniature noses
- Microelectronic technology to miniaturize



Silicon Microtechnology



- Micronoses!
- Employ Si microtechnology to reduce the cost and size
- New range of battery powered miniature e-noses
- Arrays of e-noses
- Batch fabrication
- Silicon microtechnology will not only permit the integration of a number of different sensors onto a single chip but also integration of Integrated circuitry (IC)
- ASICs for gas sensors such as MOS and QCM
- It is desirable to integrate sensor and IC on the same silicon chip to reduce the size and manufacturing costs
- Integration of sensor can pose a serious problem when combined non-standard materials, processes, and silicon CMOS
- Develop silicon micropipes, microvalves, and micropumps



Important instrument and test conditions when employing electronic nose technology

Parameter/Condition Effect

Temperature Causes significant

changes in all types

Humidity Significant variation in

many sensors

Reference gas Stable baseline signal,

reduces interference

Flow rate Sensor output varies

Odour concentration Natural variations

cause errors in sensors

Odour profile Repeatable

concentration profile

may improve

discrimination power

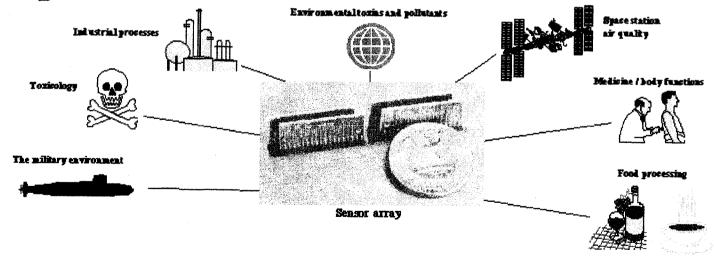
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JPL E - Nose

- ◆ STS-95 weighs 1.4 kg includes computer control
- ◆ Volume 1700 cm³ (18.5 cm x 11.5 cm x 8 cm)
- ◆ Average power 1.5 W (3 Watts peak power)
- ◆ E-Nose experiment had taken place in flight since 80% of the success criteria for ground testing had been met before the flight



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Package



- ◆ 32 Sensor head in an e-nose arrangement on 4 substrates each with 8 sensors
- ◆ Hybrid microelectronic co-fired ceramic alumina substrate
- ◆ Thick film screen printing
- ◆ A guard ring around each sensor
- ◆ 16 polymers used
- ◆ Two sensors for each polymer
- ◆ Four polymers on each substrate
- ◆ Thermistor is used to control temperature
- ◆ Gold contacts



Characteristics of sensing film response to contaminant

- Noise level
- Limit of response
- Recovery time
- Response across the polymer film array
- Linearity of response with concentration
- Deposit the film and determine the best conditions for making the film

JPL E-Nose on a Single Chip

- ❖E-Nose can be integrated with associated electronics on the same character to produce better electrical response.
- ❖Integration can be done in the same wafer level or through wafer bonding or utilizing multi-chip module carriers

JPL Packaging Reliability Concerns

- Typical mechanical fatigue
- Contamination
- **❖** E-Nose fatigue
- Media compatibility
- Device passivation and Alternative chip mounting techniques



JPL COTS E-Nose Program in Summary

- Single set of reliability testing requirements for a supplications may not be possible for evaluation of E-Nose technology
- ❖ Finding a common denominator and standardized testing based on the E-Nose key failure mechanisms are valuable to user community
- User can carryout any additional reliability testing needed for their application
- Standardized test methodology will reduce unclear communication between users and suppliers

JPL COTS E-Nose Program in Summary

- ❖ Start with high volume COTS type E-Nose components that have potential for high reliability application
- ❖ Their availability and lower cost, a large number of these components can be tested to generate statistically meaningful reliability data.
- ❖ JPL has initiated COTS E-Nose Program with objectives of understanding quality and reliability assurance associated with implementation of this technology and help to build needed infrastructure.

JPL COTS E-Nose Packaging Program



- * Similar to COTS IC Packaging Program (JPL)
- ❖ To form industry-wide consortium from aerospace, military, and commercial sectors
- ❖ Emphasize development of test methodologies for characterizing reliability of COTS E-Nose
- ❖ JPL has unique applications such as international space station (ISS) and space flight experiments for astronaut habitat



Interconnections

- Wire Bonds
- Tape Automated Bonding
- Flip-chip solder based chip connection
- Direct metal deposition
- Conductive adhesives
- Laser Pantography
- Microbump approach
- Finer linewidth wirebonds





Interconnect Materials

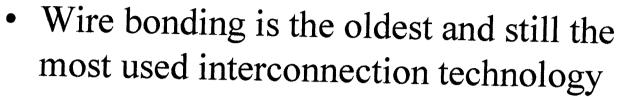


- **❖**Microstructure
- Creep Behavior
- Fracture Behavior
- Thermomechanical Fatigue Behavior

Ref.: D.R. Frear, JOM, May 1996, p.49.



Wire Bonds





- * Inexpensive
- * Well understood
- * Accessible
- Disadvantages
 - * Higher inductance
 - * Offer limited repair/rework capability
 - * Lower component densities due to large footprints on the substrate

Benefits of Advanced Packaging

- Reduction in size and weight in advanced packaging will improve component density and subsequently lead to efficient packaging.
- Great interconnection density
- Accommodate more pin counts
- Higher performance due to decrease in interconnection length
- Reliability of systems built with advanced packaging is greater, due to systematic reduction of the number of interconnections.

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Packaging of E-Noses



• Integration of E-Nose devices with any present and proposed multichip packaging is a challenging job



 Qualification and Integration of E-Nose for space applications needs advanced packaging approach

JPL Packaging of E-Nose

- Packaging of E-Nose with other system elements
- Conventional package-board-box architecture similar to soldering of IC's to PWB
- Integration of one or more E-Nose devices with interconnects and may be soldered to PCB
- Role of E-Nose Packaging technologies for the vision of Space Applications
- Qualification of E-Nose packaging for space
- Integrated E-Nose packaging for Space



Space Systems

- Qualification of the devices for space systems
- Reliability
- Battery of test methods to establish above to validate the efficacy of E-Nose
- Variety of E-Nose configurations makes difficult in terms of qualification procedures
- Building in quality E-Nose





Space Systems



- Minimization of size and weight
- Improve performance
- Enhance the awareness of E-Nose to designer of space systems
- Advanced electronics packaging
- Radiation tolerant microcircuits



Qualification

◆Suitability of technology for a particular application



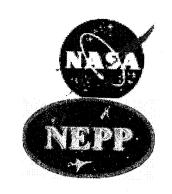
- ◆Inadequacy of military standards
- ◆Required amendment of standards
- ◆Risk assessment or factor
- ◆Assurance of new technologies

JPL Qualification Process

- ◆ Identification of technology failure modes
- ◆ Certainty of non-failure modes during the life of the mission
- **◆** Development of test methods for demonstrating failure modes (This is the current practice)
- **♦** Hermetic encapsulation
- **◆** Availability of Packaging support equipment
- ◆ Cost of packaging is a key acceptance criteria
- ◆ Space is one of the last place for the debut of a new technology

Environmental

- Conditions of storage
- Usage environment
- Qualification testing
- Incubation period of space electronics assemblies in various storage locations
- Thermal cycling
- Vibration due to launch and pyrotechnic release actions
- Hermetic packaging
- Outgassing products from polymeric materials



JPL Environmental (continued)

- Shock
- Humidity
- Radiation
- Corrosion
- Mismatches in TCE
- Extremes in ambient temperatures
- Thermomechanical shock



Issues to Address by Space Community



- New ways to designing systems
- Fully exploit the advantages of E-Nose and COTS E-Nose, ASIM technology
- Characterization of materials and systems at micron level
- Development of low-cost design, production, and test technologies
- Packaging and interconnection approaches
- Software interface
- Development of new product-assurance tools
- Versatile electronics package intended to drive/control microinstruments
- Software tools for data acquisition and analysis

JPL Factors Influencing Package Reliability



- Residual stress
- Stress relief
- Hermeticity
- Thermal performance
- Chemical stability
- Protection during packaging
- Shock resistance
- Electrical isolation
- Cost issues





Future Prospects of E-Nose

- Likely to make many advances in the near future
- Presence of commercial instruments to stimulate interest
- Multitype noses
- Micro and nanotype e-noses
- ASIC Electronics
- Adaptive neural networks
- Smarter, smaller, and cheaper e-noses





Conclusions

- Exciting advances in Electronic noses
- Packaging
- Reliability
- Instrumentation
- Understand olfactory mechanisms
- Fusing of Si microtechnology with e-nose
- Significant modeling efforts
- Potential market is diverse and enormous
- Fusion of microelectronics and medicine